Effect of cyclic hygrothermal aging and drying temperature on the interfacial properties of BMI/Carbon fiber composite

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1 Introduction

In aerospace and other applications, fiber-reinforced polymer matrix composites' performance in 'hot-wet' environments is an important assessment indexes [1]. Polymer-based materials are often exposed to hygrothermal environments, where water molecules can easily migrate into the polymeric matrix and reached at the interface between fiber and matrix resin [2,3]. Some physical and chemical variations generate with immersing of water. Physical changes such as micro-cracks propagation and swelling, as well as chemical changes such as hydrolysis and chemical scission can degrade properties of the materials. Also, the fiber/matrix interface can be damaged by moisture absorption [4–6] because resin is easy to absorb water which leads to volume expansion, while carbon fiber is hard to absorb water, and the resin's swelling generates stress and can cause interface de-bonding [7,8].

Stresses and micro-cracks can be caused not only during water's absorbing but also water's dry, so some researchers focused on the influence of the wet–dry cycling on composites and matrix[9,10,11].

As a macro-mechanical test method, the ILSS can be used to characterize the interfacial bond between fiber and matrix. In this work, the objective is to investigate the influence of cyclic water absorption on the interfacial properties of composites using interlaminar shear strength test. Electron microscopy was used to observe the microstructure of material changes during cyclic hygrothermal aging

2.1 Materials

The Thermoplastic resin toughened BMI resin QY9511 used in this paper was supplied by Beijing Aeronautical Manufacturing Technology Research Institute, Polyacryloniltrile based carbon fibers (CCF300) were produced by Weihai Tuozhan Fiber Co. Ltd.

The unidirectional composite panels were manufactured by autoclave molding according to curing process specified by resin manufacturers, and the fiber volume fraction (Vf) was 62±2%.

2.2 Heat-moisture treatment

Specimens with dimensions of 60×60×2 mm3 (length×width×thickness) were dried in a vacuum oven at 70℃ under a vacuum until a constant weight was achieved. Then they were put into a humidity chamber and immersed in distilled water at 71℃. Specimens were taken out as planned during heat-miosture treatment , dry with gauze and weighed using the analytical balance (with a precision of ± 0.1mg). After heat-moisture treatment for 7 days, specimens were removed from water and dried at 85℃ in a desiccators until a constant weight was obtained. These specimens were then used in the second and third absorption. In the next re-absorption steps, the percentage weight gain was determined by the original weight of dry specimen as reference. The weight values obtained from five specimens were averaged. The percent absorption content M defined as

$$M = \frac{W - W_d}{W_d} \times 100\% \qquad (1)$$

Where W is the weight of moist material and W_d is the weight of original material.

In order to examine the relationship the drying temperature's effect on the interlaminar shear strength of composites subjected to hygrothermal aging, several groups of specimens were put in the same hygrothermal environment for 7days, 14days , 42days, and then dried at 85℃ or 120℃.

2.3 Interlaminar Shear Strength Test and SEM test

In the wet-dry cyclic experiments, the Short Beam Shear samples of 20×6×2 mm^3 were performedon Instron 5500 testing machine respectively, according to the ASTM D-2344. On an average five specimens were tested and mean value of shear strength was calculated.

The fracture morphology of the composite was observed using LEO 1530 field-emission scanning electron microscopy (FESEM)

2.4 FT-IR test

Infra red spectra were acquired using a Thermo Nicolet Nexus 470 FT-IR spectrophotometer.

3 Results and discussing

3.1 Moisture absorption

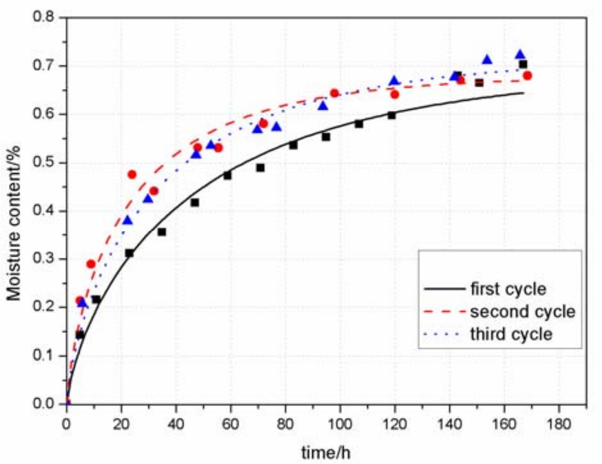


Fig 1 Cyclic moisture absorption curve for CCF300/QY9511

The 3 cycles of moisture absorption of CCF300/QY9511 panel as a function of time were shown in Fig1. The Fick's fitted curves are also plotted in Fig1, given by using Eq. (2):

$$M_t = M_\infty(1-\exp(-7.3(\frac{Dt}{b^2}))^{0.75} \quad (2)$$

where Mt is the moisture content at time t, M_∞ is the saturation level of water absorption, D is the diffusivity of the material through the thickness and b is the thickness of the specimen.

Fig1 shows that the second absorption has a similar sorption behavior to the third one, while it is different from the first absorption. Table1 also shows that the saturated moisture contents of the three absorptions are almost the same. This may be ascribed to the following two reasons: (1) voids and micro-cracks generated during the first wet-dry step. Some voids and micro-cracks growing bigger due to sorping water, and the sizes of these defects are large enough to form some channels, and water can flow out of the material through these channels thoroughly; (2) water absorption of CCF300/QY9511 composite is an "interface-dominated" phenomenon, water absorption is controlled by transport along the weak interface [14].

3.2 Interlaminar shear strength (ILSS) and wet-dry cycles

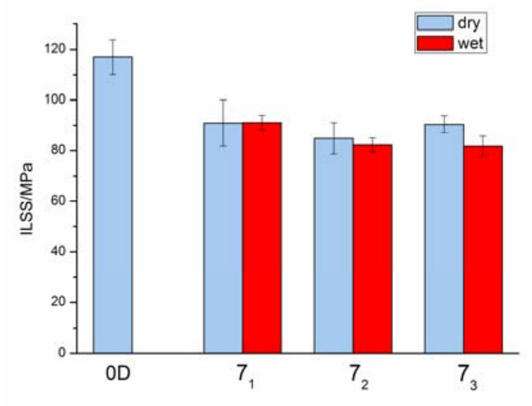
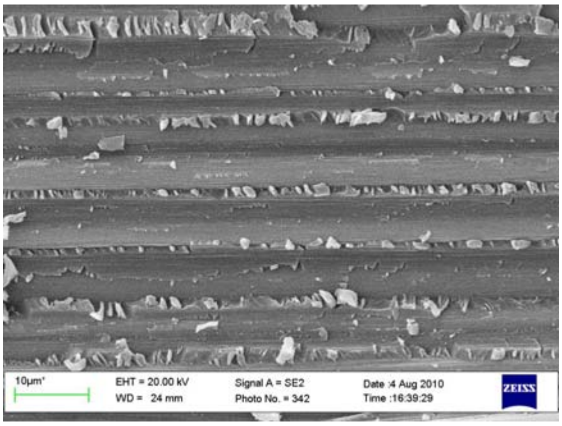


Fig 2 Interlaminar shear strength evolution during the wet–dry cycles: "7_1wet" means putting the specimens into the humidity chamber and immersed in distilled water at 71℃ for 7 days, and "7_1dry" means drying the "7_1wet" in a desiccator at 85℃ until a constant weight was obtained. By analogy, 7_2 and 7_3 have the similar means, 0D means initial state

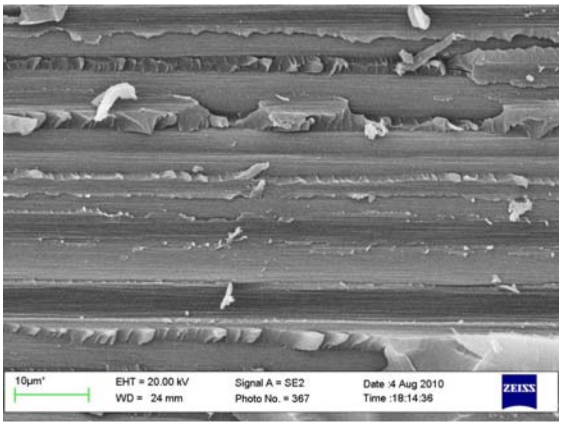
The composites shear properties are mainly decided by matrix and fiber/matrix interface [6]. So the changes of composite's performance can be obtained by the macroscopic interlaminar shear test. The short beam method is one of the simplest tests and is widely used for measuring the interlaminar shear strength (ILSS) of composites.

Fig2 shows that the ILSS values decrease when suffered with hygrothermal aging. The values loss is contributed to matrix plasticization and micro-cracking and voids. Dry specimens have a higher ILSS values than wet specimens suffering from the same aging, which is due to some physical changes, such as matrix plasticization. Network relaxation can partially recover after drying.

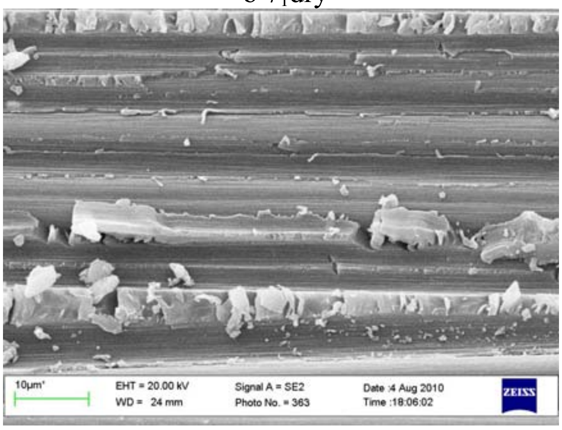
Fig3 shows the morphology of different CCF300/QY9511 composites suffered with short beam interlaminar shear damage.



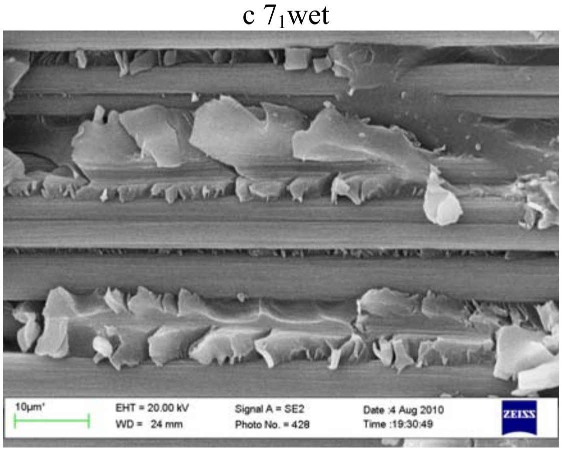
a 0D



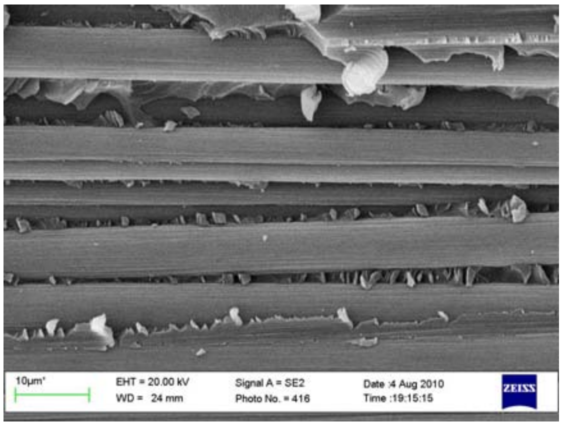
b 7_1dry



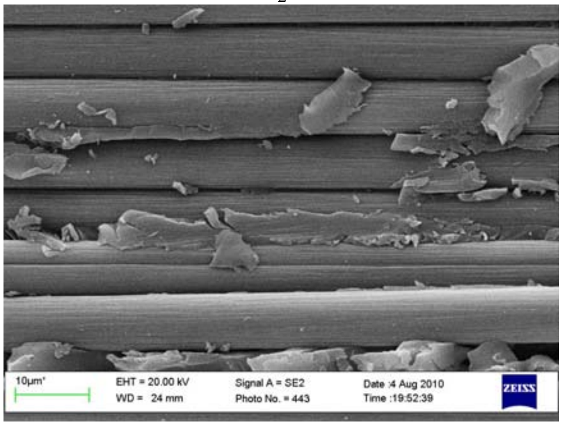
c 7_1wet



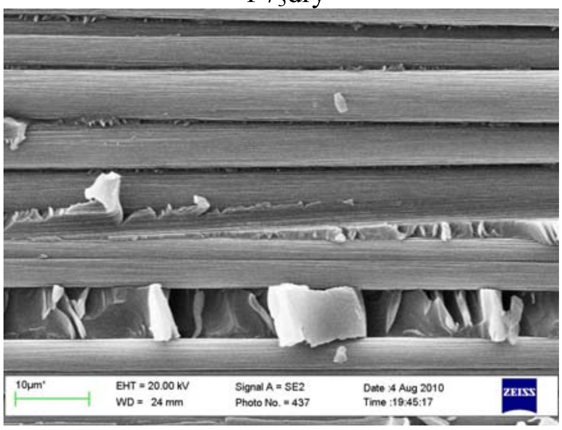
d 7_2dry



e 7_2wet



f 7_3dry



g 7_3wet

Fig3 Cross sectional area of CCF300/QY9511 materials' damage fracture

It is found that the surface of fibers is smooth with slight amount of resin adhered on the surface for "7_3wet" and "7_3dry" specimens, and the dominating deformation mechanism is de-bonding. It suggests that the interfacial bond strengths between the fiber and the matrix are poor due to hygrothermal aging, and the influence of wet-dry cycles on the composite's interface will accumulate.

In addition, it can be seen that "7_1" specimens' fibers are almost in the resin while "7_3" specimens are almost extracted from the resin and smooth. This suggests that the interfacial performance of "7_1" specimens is better than "7_3" specimens. But the result of short beam tests in Fig 2 shows that "7_1" specimens and "7_3" specimens have a close ILSS. That the properties of both matrix and fiber/matrix interface decide composites' shear properties are further proved.

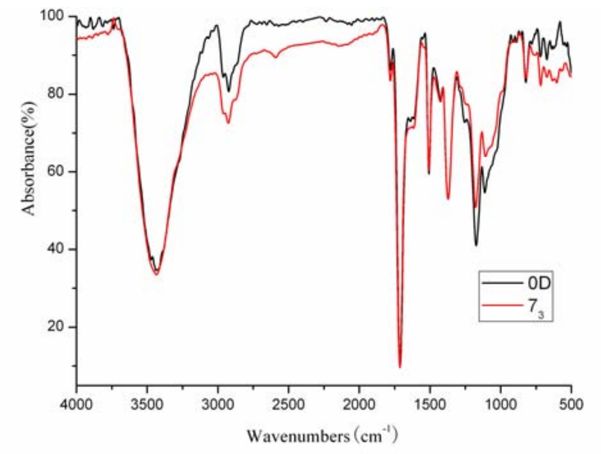


Fig3 FTIR spectra of different specimens

To examine whether heat-moisture treatment will cause any chemical structure changes of resin matrix, FTIR was used.

FTIR results showed that "0D" specimens and "7_3wet" have almost identical spectra. This suggests that chemical structure change is not a major damage factor in heat-moisture treatment especially when the treatment time is not long.

3.3 Drying temperature and ILSS

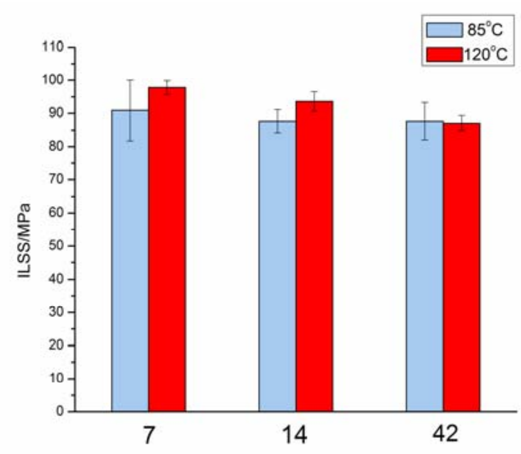


Fig4 ILSS of different dry specimens suffer from different moisture absorption days

To make sure whether drying process will lead to the decrease of composite's performance, two different temperatures were chosen to study the relationship between drying temperature and ILSS. Because the boiling point of water is 100℃,one temperature(85℃) below 100℃ and the other one(120℃) higher than 100℃ were chosen.

It could be seen that specimens suffer from heat-moisture treatment for 7days and 14days have a higher ILSS when they were dried under 120℃ than under 85℃. It is possible to hypothesize that there are two main affecting factors during the drying process: 1) the water chased out of the material, which could increase the ILSS of composites; 2) damaging effects on the composites from evaporation process. For CCF300/QY9511 composite, the first effect played a leading role. So we can see that the ILSS of specimens dried under 120℃ is above under 85℃.

When the treatment is 42 days, the damaging effects from heat-moisture treatment reach an extreme, so drying temperature can hardly influence the ILSS.

4 Conclusions

1) During the wet-dry cycles, the re-absorption of CCF300/QY9511 composite steps exhibit higher diffusivity while lower saturated moisture content.
2) SEM observations show that there are some criss-crossing voids and de-bonding formed near the fibers during the first moisture absorption, which may lead to a higher absorption rate.
3) FTIR results showed that the chemical structure had changed little.
4) It is can be inferred that there are two main affecting factors during the drying process: 1) the water is chased out of the material, 2) damaging effects on the material from water vapor.

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